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(54) Ti-Al INTERMETALLIC COMPOUND SHEET AND ITS PRODUCING METHOD

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a method for producing a Ti-Al intermetallic compound sheet easily producible with ordinary rolling mill and to provide the intermetallic compound sheet.
SOLUTION: The method consists of a rolling pressure welding process in which a laminated sheet is obtained by alternately laminating a Ti layer and an Al layer so that the main phase of an intermetallic compound becomes the two phase structure of Ti3Al and TiAl, the first solid phase diffusion heat treatment process in which, under continuously or intermittently applied pressure at the total draft of ≥3%, the laminated sheet is heated and held to a temperature less than the melting point of Al, so that Ti in the Ti layer and Al in the Al layer are reacted to form Al3Ti, to obtain the first reaction laminated sheet in which the remaining Ti layer composed of unreacted Ti and the Al3Ti layer composed of Al3Ti are laminated and the second solid phase diffusion heat treatment process in which the first reaction laminated sheet is heated and held to form a two phase structure of Ti3Al and TiAl as the main phases.

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TITLE: Titanium-aluminum group intermetallic compound board manufacture involves laminating titanium and aluminum layers alternately to form phases with intermetallic compounds, then performing solid-phase diffusion heating

PATENT-ASSIGNEE: SUMITOMO SPECIAL METALS CO LTD[SUMS]

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ABSTRACTED-PUB-NO: JP2001271127A

BASIC-ABSTRACT:

NOVELTY - A laminate is formed by roll joining of titanium (Ti) layers and aluminum (Al) layers alternately so as to form two phases with intermetallic compounds (Ti_3Al and TiAl). Solid-phase (I) diffusion heating forms Al_3Ti and a reaction laminate (i). The laminate (i) has Al_3Ti layer laminated with remaining Ti layer with unreacted Ti. Solid phase (II) diffusion heating forms a 2-phase structure.

DETAILED DESCRIPTION - A laminate is formed by roll joining of titanium (Ti) layers containing Ti and aluminum (Al) layers containing Al alternately so as to form 2 phases with main components of intermetallic compounds such as Ti_3Al and TiAl. The solid phase (I) diffusion heating forms Al_3Ti by reacting Ti of the Ti layer and Al of the Al layer and maintaining at a temperature below the melting point of Al by applying pressure continuously and intermittently at 3% or more of the total pressure, and a reaction laminate (i). The laminate (i) has Al_3Ti layer laminated with remaining Ti layer containing unreacted Ti. The solid phase (II) diffusion heating forms a 2-phase structure which makes Ti_3Al and TiAl as main phase by heating and maintaining the reaction laminate (i).

An INDEPENDENT CLAIM is also included for Ti-Al group intermetallic compound board in which almost the whole region in horizontal direction consists of Ti_3Al/TiAl lamella grain structure. The lamella grain structure has Ti_3Al/TiAl lamella, oriented almost in parallel with the board surface, as main portion.

USE - Manufacture of Ti-Al group intermetallic compound board used as lightweight heat resisting material.

ADVANTAGE - The Ti-Al group intermetallic compound board is manufactured easily

and efficiently. The board has favorable mechanical capability on its surface good practicability. The board is effectively utilized as light weight heat resisting material.

CHOSEN-DRAWING: Dwg.0/6

TITLE-TERMS: TITANIUM GROUP INTERMETALLIC COMPOUND BOARD MANUFACTURE LAMINATE
TITANIUM LAYER ALTERNATE FORM PHASE INTERMETALLIC COMPOUND PERFORMANCE SOLID PHASE DIFFUSION HEAT

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TITLE: Ti-Al INTERMETALLIC COMPOUND SHEET AND ITS PRODUCING METHOD

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ABSTRACT:

PROBLEM TO BE SOLVED: To provide a method for producing a Ti-Al intermetallic compound sheet easily producible with ordinary rolling mill and to provide the intermetallic compound sheet.

SOLUTION: The method consists of a rolling pressure welding process in which a laminated sheet is obtained by alternately laminating a Ti layer and an Al layer so that the main phase of an intermetallic compound becomes the two phase structure of Ti_3Al and TiAl, the first solid phase diffusion heat treatment process in which, under continuously or intermittently applied pressure at the total draft of

CLAIMS

[Claim(s)]
[Claim 1] The main phase of an intermetallic compound is Ti3aluminum. The rolling junction process that aluminum layer which consists of a Ti layer which consists of Ti so that it may become 2 phase organizations of TiAl, and aluminum produces the layered product by which the laminating was carried out by turns by rolling junction, Carry out heating maintenance, Ti of the aforementioned Ti layer and aluminum of the aforementioned aluminum layer are made to react to the temperature of under the melting point of aluminum, pressurizing the rate of the bottom of total pressure continuously as 3% or more, or intermittently, and it is aluminum3Ti. It is made to generate. the residual Ti layer which consists of unreacted Ti, and aforementioned aluminum3Ti from -- becoming aluminum3Ti layer With the 1st solid phase diffusion heat treatment which forms the 1st reaction layered product by which the laminating was carried out Heating maintenance of the aforementioned 1st reaction layered product is carried out. The manufacture method of the Ti-aluminum system intermetallic-compound board equipped with the 2nd solid phase diffusion heat treatment which forms 2 phase organizations which make Ti3aluminum and TiAl the main phase.
[Claim 2] Carry out heating maintenance of the aforementioned 1st reaction layered product at the temperature of less than 882 degrees C, make Ti of the aforementioned residual Ti layer, and aluminum3Ti of the aforementioned aluminum3Ti layer react in the aforementioned 2nd solid phase diffusion heat treatment, and Ti3aluminum and TiAl are made to generate. Let this Ti3aluminum be the main phase. The manufacture method of the Ti-aluminum system intermetallic-compound board indicated to the claim 1 which forms the 2nd reaction layered product to which the laminating of the TiAl layer which makes Above TiAl the main phase with a Ti3aluminum layer was carried out.
[Claim 3] The aforementioned 2nd reaction layered product The manufacture method of an Ti-aluminum system intermetallic-compound board of having indicated the 3rd solid phase diffusion heat treatment which carry out heating maintenance in alphaTi single phase temperature region of eutectoid ******** of Ti3aluminum and TiAl, and each phase of the aforementioned 2nd reaction layered product is made metamorphosing into alphaTi phase, and is cooled after that to the claim 2 which it had further.
[Claim 4] The aforementioned 2nd reaction layered product The manufacture method of the Ti-aluminum system intermetallic-compound board indicated to the claim 2 which equipped further the temperature of under the eutectoid transformation point of Ti3aluminum and TiAl, or the coexistence temperature region of alphaTi and TiAl with the 4th solid phase diffusion heat treatment which carries out heating maintenance.
[Claim 5] As the aforementioned 2nd solid phase diffusion heat treatment, it is Ti3aluminum about the aforementioned 1st reaction layered product. The manufacture method of the Ti-aluminum system intermetallic-compound board indicated to the claim 1 which performs heat treatment to which carry out heating maintenance in alphaTi single phase temperature region of eutectoid ******** of TiAl, and each phase of the aforementioned 1st reaction layered product is made to metamorphose into alphaTi phase, and is cooled after that.
[Claim 6] The aforementioned layered product is the manufacture method of the Ti-aluminum system intermetallic-compound board indicated in any 1 term of the claims 1-5 which consist of odd layers, and by which Ti layer or aluminum layer has been symmetrically arranged to a main layer.
[Claim 7] The simultaneously whole region of the direction of board thickness is . Consisting of a Ti3 aluminum/TiAl lamellae grain organization, the aforementioned lamellae grain organization did orientation to parallel mostly at the plate surface. Ti-aluminum system intermetallic-compound board which makes a Ti3 aluminum/TiAl lamellae a subject.
[Claim 8] The Ti-aluminum system intermetallic-compound board with which the laminating of the TiAl layer which makes the main phase the Ti3aluminum layer which makes a Ti3aluminum phase the main phase, and a TiAl phase was carried out to the macro target in the direction of board thickness.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] this invention relates to an Ti-aluminum system intermetallic-compound board suitable as a lightweight heat-resisting material etc., and its manufacture method.
[0002]
[Description of the Prior Art] TiAl and Ti3aluminum The Ti-aluminum system intermetallic compound which has 2 phase organizations is a material expected as a lightweight heat-resisting material, and can use the intermetallic-compound board for various uses.
[0003] That an Ti-aluminum system alloy tends to oxidize, although to manufacture an Ti-aluminum system intermetallic-compound board by the dissolution rolling-out method for carrying out the arc dissolution, casting an Ti-aluminum system alloy, and rolling out the ingot is tried, since it is lacking in ductility, the manufacture method has still stopped at the experiment stage, and the Ti-aluminum system intermetallic-compound board with practicality producible industrial now is not obtained.
[0004] Moreover, although powder-metallurgy processing which carries out compacting to the configuration which approximated the mixed-powder end of Ti powder and aluminum powder to the product configuration as the manufacture method of an Ti-aluminum system intermetallic compound and which is sintered by solid phase diffusion is also tried, expensive Ti powder is needed, and it is inferior to productivity, and is not suitable for manufacture of the plate of a large area primarily.
[0005]
[Problem(s) to be Solved by the Invention] As mentioned above, the dissolution rolling-out method and powder-metallurgy processing are unsuitable for manufacturing an Ti-aluminum system intermetallic-compound board industrially, and it has not resulted in the practical use stage.
[0006] In addition, although the method of pressing down the layered product which carried out the laminating of nickel foil and the Ti foil by turns, performing solid phase diffusion heat treatment and liquid phase diffusion heat treatment to this pressing-down plywood, and manufacturing a nickel-Ti intermetallic-compound board is indicated by JP,7-54068,A The nickel-Ti intermetallic compound and Ti3aluminum which are a single phase organization As for the Ti-aluminum system intermetallic compound which is 2 phase organizations with TiAl, organizations essentially differ. Moreover, there is no place indicated and suggested about the point of manufacturing an Ti-aluminum system intermetallic-compound board at the aforementioned official report by being made from Ti of a high-melting point and aluminum of the low melting point.
[0007] this invention is made in view of this problem, and it aims at offering the Ti-aluminum system intermetallic-compound board excellent in the manufacture method of an Ti-aluminum system intermetallic-compound board of having excelled in practicality as a industrial process, and the mechanical property of the direction of a plate surface. This purpose is attained by the following invention.
[0008]
[Means for Solving the Problem] The manufacture method of the Ti-aluminum system intermetallic-compound board of this invention As indicated to the claim 1, the main phase of an intermetallic compound is Ti3aluminum. The rolling junction process that aluminum layer which consists of a Ti layer which consists of Ti so that it may become 2 phase organizations of TiAl, and aluminum produces the layered product by which the laminating was carried out by turns by rolling junction, Carry out heating maintenance, Ti of the aforementioned Ti layer and aluminum of the aforementioned aluminum layer are made to react to the temperature of under the melting point of aluminum, pressurizing the rate of the bottom of total pressure continuously as 3% or more, or intermittently, and it is aluminum3Ti. It is made to generate. The 1st solid phase diffusion heat treatment which forms the 1st reaction layered product to which the laminating of the aluminum3Ti layer which consists of a residual Ti layer which consists of unreacted Ti, and aforementioned aluminum3Ti was carried out, Heating maintenance of the aforementioned 1st reaction layered product is carried out, and it is Ti3aluminum. It has the 2nd solid phase diffusion heat treatment which forms 2 phase organizations which make TiAl the main phase.
[0009] According to this invention, since a layered product is produced by rolling junction, if the plywood of a tabular with a large surface area can be easily obtained with the usual rolling facility and it lengthens, the Ti-aluminum system intermetallic-compound board of a large area can be easily manufactured by performing the 1st solid phase diffusion heat treatment and the 2nd solid phase diffusion heat treatment. by the way, aluminum of aluminum layer and Ti of Ti layer -- reacting -- aluminum3Ti Ti and aluminum besides the opening (void) according to a Kirkendall effect in case it generates -- and -- Based

on a difference of each crystal structure of aluminum3Ti, about 3% of volume decrease arises, a lot of openings occur, and in being remarkable, ablation arises. For this reason, even if it only performs solid phase diffusion heat treatment at the temperature of under the melting point of aluminum, a reaction is suppressed for an opening and unreacted aluminum comes to remain. When there is survival aluminum, in case it is the 2nd solid phase diffusion heat treatment, Survival aluminum flows out of a layered product, and an Ti-aluminum system intermetallic compound is not obtained. Heating maintenance of the aforementioned layered product is carried out at the temperature of under the melting point of aluminum, pressurizing the rate of the bottom of total pressure 3% or more continuously [it is desirable and] as 5% or more, or intermittently that the rate of volume of a layered product should be reduced 3% or more in the case of the 1st solid phase diffusion heat treatment which makes aluminum3Ti generate in this invention. Consequently, it is a residual Ti layer, suppressing or preventing generation of an opening. An aluminum3Ti layer can form easily the 1st reaction layered product by which the laminating was carried out. since aluminum is not substantially contained in this 1st reaction layered product, in the 2nd solid phase diffusion heat treatment after the 1st solid phase diffusion heat treatment, heating maintenance can be carried out at the elevated temperature beyond the melting point of aluminum -- the Ti-aluminum system intermetallic-compound board which consists of 2 phase organizations which make a Ti3aluminum phase and a TiAl phase the main phase can be manufactured easily and efficiently

[0010] Moreover, as indicated to the claim 2, the aforementioned 2nd solid phase diffusion heat treatment carries out heating maintenance of the aforementioned 1st reaction layered product at the temperature of less than 882 degrees C, and describes it above with Ti of the aforementioned residual Ti layer. aluminum3Ti of an aluminum3Ti layer is made to react. Ti3aluminum and TiAl are made to generate. The TiAl layer which makes Above TiAl the main phase with the Ti3aluminum layer which makes this Ti3aluminum the main phase can consider as heat treatment which forms the 2nd reaction layered product by which the laminating was carried out. According to the 2nd solid phase diffusion heat treatment indicated to this claim 2, since a heating retention temperature is less than 882 degrees C, Survival Ti (alphaTi) metamorphoses and does not grow up to be betaTi from which the crystal structure completely differs. Therefore, have held the orientation of the specific crystal face to parallel mostly at the plate surface. The 2nd reaction layered product of the structure where the laminating of a Ti3aluminum layer and the TiAl layer was carried out in layers can be obtained easily. This 2nd reaction layered product can be used as an Ti-aluminum system intermetallic-compound board with which it has the layer structure, and the crystal orientation of each class has a good mechanical property because it is parallel to a plate surface in general.

[0011] Moreover, as indicated to the claim 3, the amount of openings under organization can be decreased by performing the back-cooled 3rd solid phase diffusion heat treatment to which carried out heating maintenance of the aforementioned 2nd reaction layered product in alphaTi single phase temperature region, and each phase of the aforementioned 2nd reaction layered product was made to metamorphose into alphaTi phase. Moreover, orientation was mostly carried out to parallel over the simultaneously whole region of the direction of board thickness at the plate surface. Let a Ti3 aluminum/TiAl lamellae be a subject. The Ti-aluminum system intermetallic-compound board which consists of a Ti3 aluminum/TiAl lamellae grain organization can be obtained easily. This intermetallic-compound board is excellent in the mechanical property of the crystal structure, therefore the direction of a plate surface.

[0012] Moreover, as indicated to the claim 4, it is the aforementioned 2nd reaction layered product. An Ti-aluminum system intermetallic-compound board with the good mechanical property which decreased the opening under organization can be obtained easily, holding the orientation and the lamellar structure of a crystal by performing the 4th solid phase diffusion heat treatment which carries out heating maintenance to the temperature of under the eutectoid transformation point of Ti3aluminum and TiAl, or the coexistence temperature region of alphaTi and TiAl.

[0013] Moreover, as the aforementioned 2nd solid phase diffusion heat treatment, as indicated to the claim 5 By carrying out heating maintenance of the aforementioned 1st reaction layered product in alphaTi single phase temperature region, making each phase of the aforementioned 1st reaction layered product metamorphose into alphaTi phase, and cooling after that Since Survival Ti once metamorphosed into betaTi, the orientation of a crystal randomized, therefore eutectoid transformation generated. Although the orientation of a Ti3 aluminum/TiAl lamellae is also randomized The Ti-aluminum system intermetallic-compound board which has the Ti3 aluminum/TiAl lamellae grain structure can be manufactured easily.

[0014] Moreover, as indicated to the claim 6, by making the aforementioned layered product into the structure which consists of odd layers by which Ti layer or aluminum layer has been arranged symmetrically to a main layer, the heat deformation resulting from the coefficient-of-thermal-expansion difference of Ti and aluminum can be prevented, the manufacture trouble resulting from heat deformation can be prevented, and productivity can be raised. In this case, since all aluminum layers will be in the state where it was pinched by Ti layer, by using an outermost layer of drum as Ti layer, aluminum of all aluminum layers is made to react reasonable in the case of the 1st solid phase diffusion heat treatment. aluminum3Ti can be made to be able to generate and survival of unreacted aluminum can be prevented. Moreover, even if unreacted aluminum should remain, the defluxion can be prevented in the case of elevated-temperature heat treatment, and the manufacture yield of an expected Ti-aluminum system intermetallic-compound board can be raised.

[0015] For the Ti-aluminum system intermetallic-compound board of this invention, as indicated to the claim 7, the simultaneously whole region of the direction of board thickness is substantially. Consisting of a Ti3 aluminum/TiAl lamellae grain organization, the aforementioned lamellae grain organization did orientation to parallel mostly at the plate surface. Since a Ti3 aluminum/TiAl lamellae is made into a subject, it excels in the mechanical property of the direction of a plate surface.

[0016] Moreover, since the laminating of the TiAl layer which makes the main phase the Ti3aluminum layer which makes a Ti3aluminum phase the main phase, and a TiAl phase is carried out to a macro target in the direction of board thickness as indicated to the claim 8, other Ti-aluminum system intermetallic-compound boards of this invention are equipped with a good

mechanical property as compared with the conventional intermetallic-compound board with the homogeneous interior of board thickness.

[0017]

[Embodiments of the Invention] The Ti-aluminum system intermetallic-compound board hereafter applied to this invention obtained by the manufacture method of the Ti-aluminum system intermetallic-compound board of this invention and this manufacture method is explained in detail.

[0018] In order to enforce the manufacture method of this invention, aluminum layer which consists of a Ti layer which consists of Ti according to a rolling junction process, and aluminum first produces the layered product by which the laminating was carried out by turns. Rolling junction of the proper number of sheets is carried out for Ti sheet metal and aluminum sheet metal in piles, the aforementioned rolling junction process obtains the laminating material of two or more layers, and the aforementioned laminating material is suitably doubled in several [-fold], and it carries out rolling junction so that Ti layer and aluminum layer may be arranged further by turns. What is necessary is just to perform the rolling reduction per rolling at about 25 - 50% preferably 20 to 60% that what is necessary is just to perform rolling junction between the colds. In order that after rolling junction may raise the bonding strength of each class, it is good that for temperature below the melting point of aluminum to be performed in inert gas atmosphere, such as argon gas, and it is made to perform homogenizing for several minutes - about about ten minutes for a laminating material or its jointing material at about 600 degrees C.

[0019] Since a combustion reaction will come to arise in the case of the below-mentioned homogenizing in a rolling junction process if thickness is too thin, the thickness of each class of the aforementioned layered product is 2 micrometers. It is good above to make it 5 micrometers or more preferably. On the other hand, since solid phase diffusion heat treatment mentioned later will take a long time and it will come to spoil industrial productivity if too thick, it is 50 micrometers. It is 30 micrometers preferably hereafter. It is good to make it below. In addition, the thickness of each class of the aforementioned layered product can be adjusted by cold-rolling suitably to the back in the middle of a rolling junction process.

[0020] Although the number of each class of Ti layer of the aforementioned layered product and aluminum layer can be set up arbitrarily, it sets in the thickness direction (the direction of a laminating) of a layered product. It is necessary to set up Ti-aluminum composition so that 2 phase organizations of Ti3aluminum and TiAl may be obtained. as the average composition from which 2 phase organizations are obtained -- at% -- Ti: -- it is about (remainder aluminum) 60 - 52% preferably 65 to 52%

[0021] But as for the number of layers of the aforementioned layered product, it is good to consider as odd number preferably and to make it Ti layer come to an outermost layer of drum. Thus, heat deformation of the curvature which originated the main layer in the coefficient-of-thermal-expansion difference on the occasion of the below-mentioned solid phase diffusion heat treatment by arranging Ti layer and aluminum layer symmetrically [with the thickness direction] as a center can be prevented. Moreover, since aluminum layer is surely pinched by Ti layer, it comes to react easily certainly with Ti in the case of the below-mentioned 1st solid phase diffusion heat treatment, and it can prevent survival of unreacted aluminum. Moreover, even if unreacted aluminum should remain, the defluxion can be prevented in the case of elevated-temperature heat treatment.

[0022] Next, as for the layered product produced by the rolling junction process, the 1st solid phase diffusion heat treatment is performed. this 1st solid phase diffusion heat treatment -- the temperature of under the melting point of aluminum -- desirable -- 630-500 degrees C -- more -- desirable -- the temperature of about 620-550 degrees C -- it is -- 40min - until hold about 10 hrs, it makes Ti of Ti layer of the aforementioned layered product, and aluminum of aluminum layer react and aluminum is lost fundamentally It is the processing which makes aluminum3Ti generate. For example, when setting average composition to Ti-45at%aluminum, Ti reacts as all aluminum by the following formula. aluminum3Ti generates and unreacted Ti remains. The 1st reaction layered product the aluminum3Ti layer which consists of Ti layer and the aluminum3Ti phase which consist of survival Ti with the 1st solid phase diffusion heat treatment after all carried out [the layered product] the laminating is obtained. 55Ti+45aluminum->40Ti+15aluminum3Ti[0023] By the way, Ti, aluminum, and aluminum3Ti It is aluminum3Ti if a volume change is calculated in consideration of a lattice constant from the crystal structure. A volume decrease happens at the reaction at the time of generating, and the percentage reduction becomes 3.2% in the case where it is the aforementioned instantiation composition. Along with a residual Ti layer, a lot of openings (void) are formed of the aforementioned volume change besides a Kirkendall effect, and the aforementioned diffusion reaction comes to be prevented. Moreover, in being remarkable, it also invites ablation of a layer. The 3.2% of the aforementioned rates of a volume decrease is Ti3aluminum although it is the case of Ti-45at%aluminum. The component range from which 2 phase organizations with TiAl are obtained is considered that about 3 - 4% of opening is formed in general. In addition, since the laminating of aluminum layer and the Ti layer is carried out in the thickness direction, the decrement of the aforementioned rate of volume can be considered as a percentage reduction of the direction of board thickness.

[0024] For this reason, at this invention, it is aluminum3Ti in the case of the 1st solid phase diffusion heat treatment. In case it generates, a layered product is preferably pressed down for the rate of the bottom of total pressure continuously or intermittently as 5% or more 3% or more that the opening formed should be eliminated. An opening is eliminated by this and it is aluminum3Ti. A generation reaction is promoted. You may give preferably the lightly pressurizing [several % or about about ten% of] at intervals of several minutes or dozens of minutes several times or more, or may make it always add the compressive load of about 10-70 MPas to a layered product in the middle of heat treatment at a layered product as the pressing-down method in the case of the aforementioned 1st solid phase diffusion heat treatment. In addition, a rolling reduction (%) means a (board thickness decrement) / (initial board thickness) x100.

[0025] Next, it is Ti3aluminum to the 1st reaction layered product obtained by the aforementioned 1st solid phase diffusion heat treatment. The 2nd solid phase diffusion heat treatment which forms 2 phase organizations which make TiAl the main phase is

performed. If the previous example of composition explains, the reaction in this 2nd solid phase diffusion heat treatment can be expressed with the following formula. In addition, at this reaction, the volume of the whole phase becomes about 2% of increase. 40Ti+15aluminum3Ti ->5Ti3aluminum+40TiAl[0026] Carry out heating maintenance of the aforementioned 1st reaction layered product at the temperature of less than 882 degrees C, make Ti of the aforementioned residual Ti layer, and aluminum3Ti of the aforementioned aluminum3Ti layer react as this 2nd solid phase diffusion heat treatment, and Ti3aluminum and TiAl are made to generate. this -- The TiAl layer which makes Above TiAl the main phase with the Ti3aluminum layer which makes Ti3aluminum the main phase can take heat treatment which forms the 2nd reaction layered product by which the laminating was carried out.
[0027] the feature of this heat treatment -- Ti-aluminum of drawing 1 -- duality -- it is in the point that the 2 aforementioned phase organizations can be obtained, without making Survival Ti (alphaTi) metamorphose into betaTi so that I may be understood from T1 line described into the system state diagram If the crystal structure metamorphoses into remarkably different betaTi from alphaTi, although the orientation of a crystal will be confused and grain growth will be caused and made big and rough, 2 phase organizations can be obtained maintaining the orientation (many being parallel mostly at the plate surface.) of the crystal of alphaTi without making crystal grain turn big and rough according to this heat treatment. And in order for a Ti3aluminum phase to generate from the portion which mainly has Survival Ti and to generate an AlTi phase from the portion of aluminum3Ti, it is on a macro target. The 2nd reaction layered product of the layer structure in which the Ti3aluminum layer and the TiAl layer carried out the laminating can be obtained. That is, this layered product becomes good [the mechanical property of the direction of a plate surface] for the orientation of the layer structure and crystal face. In addition, this 2nd reaction layered product makes one mode of the Ti-aluminum system intermetallic compound of this invention.
[0028] It is desirable to perform heating maintenance of the aforementioned 2nd solid phase diffusion heat treatment in a vacuum for antioxidizing. Moreover, although what is necessary is just to set it as less than 882 degrees C in order to make it alphaTi not metamorphose into betaTi, when the stability of processing is taken into consideration, it is preferably good [a heating retention temperature] to make [870 degrees C or less] it more preferably 860 degrees C or less. Moreover, when the efficiency of solid phase diffusion is taken into consideration, it is desirable to set it as 800 degrees C or more. What is necessary is just to set the heating holding time to about 15-25 hrs, when carrying out heating maintenance at the temperature of 800 degrees C or more.
[0029] In this invention, the 3rd solid phase diffusion heat treatment which carry out heating maintenance of the 2nd reaction layered product obtained by the aforementioned 2nd solid phase diffusion heat treatment further in alphaTi single phase temperature region, and each phase of the aforementioned 2nd reaction layered product is made to once metamorphose into alphaTi phase, and is cooled after that can be performed. The upper limit should just be temperature which betaTi phase does not produce that heating temperature should just be temperature from which alphaTi single phase organization is obtained in short, without the liquid phase arising. It is desirable to perform heating maintenance in a vacuum for antioxidizing, and about 5-15 hrs are desirable [the holding time] so that big and rough-ization of crystal grain may not arise while the holding time makes all organizations metamorphose into alphaTi single phase organization.
[0030] By performing this 3rd solid phase diffusion heat treatment, as T2 line of drawing 1 shows Once metamorphosing into alphaTi, in case an eutectoid transformation point (1118 degrees C) is passed, eutectoid transformation is started, and it is Ti3aluminum. Since TiAl serves as a lamellae grain organization stratified in micro and the orientation of many lamellaes is moreover parallel mostly at a plate surface, The Ti-aluminum system intermetallic-compound board which has the mechanical property which was excellent in the direction of a plate surface can be obtained. Moreover, it also sets, when a part of intermediate-reaction product (for example, aluminum2Ti) and unreacted Ti remain in the 2nd reaction layered product, and it is completely Ti3aluminum about these. The opening which could be made to react to TiAl and remained can be decreased or vanished more, and the quality of an intermetallic compound can be raised. In addition, although pro-eutectoid [TiAl] (gamma) generates according to the fall of temperature from alphaTi in hypereutectoid composition, it is confirmed by this invention person that this gamma phase deposits in the shape of a lamellae.
[0031] As heat treatment to the aforementioned 2nd reaction layered product, as T3 line of not only the above-mentioned 3rd solid phase diffusion heat treatment but drawing 1 shows, it is Ti3aluminum about the 2nd reaction layered product. The 4th solid phase diffusion heat treatment which carries out heating maintenance at the temperature of under an eutectoid transformation point (1118 degrees C) with TiAl or the coexistence temperature region of alphaTi and TiAl (gamma) can also be performed. Since a heating retention temperature has the good higher one from promotion of a reaction, as for a minimum, considering as about 1000 degrees C is more desirably desirable 960 degrees C. But since the coexistence temperature region with gamma reaches to an elevated-temperature field as alphaTi, it is desirable to process at the temperature of under an eutectoid transformation point from big and rough-ized prevention of crystal grain and a viewpoint of maintenance of a macro target laminated structure. It is desirable to also perform heating maintenance in this 4th solid phase diffusion heat treatment in a vacuum, and about 10-25 hrs of the holding time are desirable so that big and rough-ization of crystal grain may not arise. The quality intermetallic compound to which an intermediate-reaction product etc. did not arise, and the opening also decreased or disappeared with this 4th solid phase diffusion heat treatment, maintaining the layer structure of the 2nd reaction layered product and the orientation of the direction of a plate surface of a crystal to some extent can be obtained.
[0032] The above 3rd and the 4th solid phase diffusion heat treatment are the Ti3aluminum phase obtained by performing the 2nd solid phase diffusion heat treatment. Heat treatment to the 2nd reaction layered product which makes a TiAl phase the main phase is shown. On the other hand, as 2nd solid phase diffusion heat treatment, without carrying out heating maintenance of the 1st reaction layered product at the temperature of less than 882 degrees C Heat treatment cooled after heating maintenance at the temperature of alphaTi single phase temperature region of eutectoid ******** of Ti3aluminum and TiAl (it is called alpha heat treatment.) ****** -- good -- the [or] -- 1 reaction layered product -- under an eutectoid transformation point or alpha -- heat

treatment (it is called beta heat treatment.) cooled after heating maintenance at the temperature of the coexistence temperature region of Ti and gamma You may carry out. Although the target reaction layered products differ, as a view of the heat treatment method, alpha heat treatment is equivalent to the aforementioned 3rd solid phase diffusion heat treatment, and beta heat treatment is heat treatment corresponding to the aforementioned 4th solid phase diffusion heat treatment, and it can set it up like the above 3rd and the 4th solid phase diffusion heat treatment about the heating temperature and time. But in alpha heat treatment, it is aluminum3Ti. It is necessary to carry out heating maintenance at the temperature of less than 1387 degrees C so that it may not become the liquid phase.

[0033] It is Ti3aluminum although the orientation of a crystal parallel to a plate surface will also be spoiled since in the aforementioned alpha heat treatment and beta heat treatment crystal grain turns big and rough and Survival Ti (alphaTi phase) once metamorphoses into betaTi phase. The Ti-aluminum system intermetallic-compound board which has 2 phase organizations which make TiAl the main phase can be obtained easily.

[0034] Hereafter, although an example explains this invention further, this invention is not interpreted in limitation according to this example.

[0035]

[Example] Example 1 (1) After deleting coarsely the front face of a rolling junction process unalloyed ti board and a pure aluminum board using a metallic brush, the deleted fields were piled up, it cold-rolled at 30% of rolling reductions, and the two-layer material to which 600 degrees C and homogenizing for 10 minutes were performed in argon atmosphere, and both layers stuck the composite to which the pressure welding of Ti layer and the aluminum layer was carried out was obtained after that. The six-layer material was obtained by making three sheets pile up mutually, carrying out rolling junction, and carrying out homogenizing of this two-layer material so that Ti layer and aluminum layer may lap by turns. The layered product which consists of 55 layers so that Ti layer and aluminum layer may finally pile up by turns and both outermost layers of drum may turn into Ti layer was produced. At that time, board thickness was set to Ti board:0.25mm and aluminum board:0.20mm so that average composition of a layered product might serve as Ti-45.5at%aluminum. moreover, the average thickness of Ti layer of the layered product finally obtained -- 17 micrometers the average thickness of aluminum layer -- 13 micrometers it was .

[0036] (2) A total of one hr heating maintenance was carried out at 600 degrees C to the layered product of the 1st solid phase diffusion heat treatment aforementioned 55 layers. Under the present circumstances, as shown in drawing 2 , hot rolling of 10 - 3% of rolling reductions was performed 6 times at intervals of 10 minutes. It gazed at the macro target cross-section organization of the 1st reaction layered product obtained by this heat treatment by SEM. The result is shown in drawing 3 . Since it was confirmed from analysis by EPMA that aluminum has reacted completely, it turns out that the layer observed is a residual Ti layer which consists of an aluminum3Ti layer which consists of aluminum3Ti, and unreacted Ti. Moreover, the linear opening extended in the direction of a plate surface was accepted.

[0037] (3) 20hr maintenance of the 1st reaction layered product of the 2nd solid phase diffusion heat treatment above was carried out at 850 degrees C in the vacuum. Under the present circumstances, in order to prevent that heat deformation (curvature) arises in a layered product, the weight of the piece of stainless steel was laid in the 1st reaction layered product through the ceramic board. The compressive stress concerning the aforementioned layered product was about 3 kPas. The aforementioned ceramic board is for preventing the reaction of weight and the 1st reaction layered product. It gazed at the cross-section organization of the 2nd reaction layered product obtained by this heat treatment by SEM. The result is shown in drawing 4 . The 2nd reaction layered product is mainly a TiAl phase. Although the structure where the laminating of the Ti3aluminum phase was carried out in the thickness direction was shown, Tl of a minute amount and aluminum2Ti remained, and the detailed opening was observed.

[0038] (4) In the vacuum, at 1300 degrees C, 10hr maintenance was carried out and the 3rd solid phase diffusion heat treatment and the 2nd reaction layered product of the 4th solid phase diffusion heat treatment above were annealed (the 3rd solid phase diffusion heat treatment). It gazed at the micro cross-section organization of the intermetallic-compound board obtained by this heat treatment by SEM. The result is shown in drawing 5 . As shown in drawing 5 , TiAl (gamma) / Ti3aluminum (alpha 2) lamellar organization grain the lamellae carried out [grain] orientation to the plate surface mostly at parallel were observed. Moreover, about an opening, the linear opening was lost as compared with the 2nd reaction layered product, and there were also few amounts of openings. On the other hand, the aforementioned 2nd reaction layered product was annealed after 10hr maintenance at 1100 degrees C in the vacuum (the 4th solid phase diffusion heat treatment). It gazed at the macro target cross-section organization of the intermetallic-compound board obtained by this heat treatment by SEM. This observation result is shown in drawing 6 . From drawing 6 , although the laminated structure of the direction of board thickness collapsed rather than the 2nd reaction layered product, the laminated structure was maintained as a whole by the macro target. Moreover, about the opening, although observed comparatively mostly near the front face, only the detailed opening was observed inside.

[0039] Example 2 (1) The layered product which consists of 55 layers was produced like the rolling junction process aforementioned example 1.

[0040] (2) To the layered product of the 1st solid phase diffusion heat treatment aforementioned 55 layers, the compressive stress of 50MPa(s) was added in the vacuum, and 5hr heating maintenance was carried out at 600 degrees C.

[0041] (3) 20hr maintenance of the 2nd solid phase diffusion heat treatment length, then the 1st reaction layered product was carried out at 850 degrees C. Under the present circumstances, it was made to decrease from 50MPa(s) to 10MPa(s), and compressive stress was held by after that 10MPa. Mean compressive stress was 22MPa(s). It was the grade as which the 2nd reaction layered product obtained as a result is making the layer structure the macro target, and most openings are not observed, but few linear openings are regarded locally. Moreover, the phase observed by EPMA analysis is Ti, aluminum3Ti, and aluminum2Ti in part, although the main phase was with Ti3aluminum and TiAl. It accepted and the reaction was slow as

compared with the 2nd reaction layered product of the 1st example. This is Ti and Ti3aluminum. It is guessed because the increase in volume produced in case two phases generate was suppressed with the compressive load. Therefore, the stage of the 1st solid phase diffusion heat treatment is enough as addition of a compressive load, and it is effective to stop to addition of the light load of a grade which prevents curvature generating of a layered product in the 2nd solid phase diffusion heat treatment.
[0042] (4) The 2nd reaction layered product of the 3rd solid phase diffusion heat treatment above was annealed after 10hr maintenance at 1300 degrees C in the vacuum. As a result of carrying out organization observation of the cross section of the obtained layered product in SEM, a full lamellar organization is observed, the intermetallic-compound board which whose amount of openings is very also a minute amount, and was excellent in soundness is obtained, and it is ****.
[0043]
[Effect of the Invention] According to the manufacture method of this invention, using the usual rolling facility, an Ti-aluminum system intermetallic-compound board can be manufactured easily and efficiently, and it excels as the industrial manufacture method. Moreover, the Ti-aluminum system intermetallic-compound board of this invention has the good mechanical property of the direction of a plate surface, and it is excellent in practicality.

[Translation done.]

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(54)【発明の名称】　Ｔｉ－Ａｌ系金属間化合物板およびその製造方法

(57)【要約】

【課題】　通常の圧延設備によって容易に製造することができるＴｉ－Ａｌ系金属間化合物板の製造方法およびその金属間化合物板を提供する。

【解決手段】　金属間化合物の主相がTi_3Alと$TiAl$の２相組織となるようにＴｉからなるＴｉ層とＡｌからなるＡｌ層とが交互に積層された積層体を圧延接合により作製する圧延接合工程と、全圧下率を３％以上として連続的あるいは間欠的に加圧しながらＡｌの融点未満の温度に加熱保持し、前記Ｔｉ層のＴｉと前記Ａｌ層のＡｌとを反応させてAl_3Tiを生成させ、未反応のＴｉからなる残存Ｔｉ層と前記Al_3TiからなるAl_3Ti層とが積層された第１反応積層体を形成する第１固相拡散熱処理と、前記第１反応積層体を加熱保持してTi_3Alと$TiAl$とを主相とする２相組織を形成する第２固相拡散熱処理とを備える。

【特許請求の範囲】

【請求項１】　金属間化合物の主相がＴｉ$_{3}$Ａｌ とＴｉＡｌの２相組織となるようにＴｉからなるＴｉ層とＡｌからなるＡｌ層とが交互に積層された積層体を圧延接合により作製する圧延接合工程と、
全圧下率を３％以上として連続的あるいは間欠的に加圧しながらＡｌの融点未満の温度に加熱保持し前記Ｔｉ層のＴｉと前記Ａｌ層のＡｌとを反応させてＡｌ$_{3}$Ｔｉ を生成させ、未反応のＴｉからなる残存Ｔｉ層と前記Ａｌ$_{3}$Ｔｉ からなるＡｌ$_{3}$Ｔｉ層 とが積層された第１反応積層体を形成する第１固相拡散熱処理と、
前記第１反応積層体を加熱保持して Ｔｉ$_{3}$ＡｌとＴｉＡｌとを主相とする２相組織を形成する第２固相拡散熱処理とを備えたＴｉ－Ａｌ系金属間化合物板の製造方法。

【請求項２】　前記第２固相拡散熱処理では、前記第１反応積層体を８８２℃未満の温度に加熱保持して前記残存Ｔｉ層のＴｉと前記Ａｌ$_{3}$Ｔｉ層のＡｌ$_{3}$Ｔｉとを反応させてＴｉ$_{3}$ＡｌとＴｉＡｌとを生成させ、このＴｉ$_{3}$Ａｌを主相とする Ｔｉ$_{3}$Ａｌ層と前記ＴｉＡｌを主相とするＴｉＡｌ層とが積層された第２反応積層体を形成する請求項１に記載したＴｉ－Ａｌ系金属間化合物板の製造方法。

【請求項３】　前記第２反応積層体を Ｔｉ$_{3}$ＡｌとＴｉＡｌの共析変態点超のαＴｉ単相温度域で加熱保持して前記第２反応積層体の各相をαＴｉ相に変態させ、その後冷却する第３固相拡散熱処理をさらに備えた請求項２に記載したＴｉ－Ａｌ系金属間化合物板の製造方法。

【請求項４】　前記第２反応積層体を Ｔｉ$_{3}$ＡｌとＴｉＡｌとの共析変態点未満あるいはαＴｉとＴｉＡｌとの共存温度域の温度に加熱保持する第４固相拡散熱処理をさらに備えた請求項２に記載したＴｉ－Ａｌ系金属間化合物板の製造方法。

【請求項５】　前記第２固相拡散熱処理として、前記第１反応積層体をＴｉ$_{3}$Ａｌ とＴｉＡｌの共析変態点超のαＴｉ単相温度域で加熱保持して前記第１反応積層体の各相をαＴｉ相に変態させる熱処理を行い、その後冷却する請求項１に記載したＴｉ－Ａｌ系金属間化合物板の製造方法。

【請求項６】　前記積層体は、奇数層からなり、中心層に対してＴｉ層あるいはＡｌ層が対称に配置された請求項１～５のいずれか１項に記載したＴｉ－Ａｌ系金属間化合物板の製造方法。

【請求項７】　板厚方向のほぼ全域が実質的に Ｔｉ$_{3}$Ａｌ／ＴｉＡｌラメラ粒組織からなり、前記ラメラ粒組織は板面にほぼ平行に配向した Ｔｉ$_{3}$Ａｌ／ＴｉＡｌラメラを主体とするＴｉ－Ａｌ系金属間化合物板。

【請求項８】　Ｔｉ$_{3}$Ａｌ相を主相とするＴｉ$_{3}$Ａｌ層とＴｉＡｌ相を主相とするＴｉＡｌ層とが板厚方向にマクロ的に積層されたＴｉ－Ａｌ系金属間化合物板。

【発明の詳細な説明】

【０００１】
【発明が属する技術分野】本発明は、軽量耐熱材料等として好適なＴｉ－Ａｌ系金属間化合物板およびその製造方法に関する。

【０００２】
【従来の技術】ＴｉＡｌおよびＴｉ$_{3}$Ａｌ の２相組織を有するＴｉ－Ａｌ系金属間化合物は軽量耐熱材料として期待される材料であり、その金属間化合物板は様々な用途に利用可能である。

【０００３】Ｔｉ－Ａｌ系合金をアーク溶解し、鋳造し、その鋳塊を圧延する溶解圧延法によってＴｉ－Ａｌ系金属間化合物板を製造することが試みられているが、Ｔｉ－Ａｌ系合金は酸化し易く延性に乏しいため、その製造方法は未だ実験段階に止まっており、現在のところ工業的生産可能な実用性のあるＴｉ－Ａｌ系金属間化合物板は得られていない。

【０００４】また、Ｔｉ－Ａｌ系金属間化合物の製造方法として、Ｔｉ粉末とＡｌ粉末との混合粉末を製品形状に近似した形状に圧粉成形して固相拡散によって焼結する粉末冶金法も試みられているが、高価なＴｉ粉末を必要とし、生産性に劣り、そもそも大面積の板材の製造には適さない。

【０００５】
【発明が解決しようとする課題】上記のように、溶解圧延法も粉末冶金法も、Ｔｉ－Ａｌ系金属間化合物板を工業的に製造するには不適当であり、実用段階に至っていない。

【０００６】なお、特開平７－５４０６８号公報には、Ｎｉ箔とＴｉ箔とを交互に積層した積層体を圧下し、この圧下積層材に固相拡散熱処理、液相拡散熱処理を施してＮｉ－Ｔｉ金属間化合物板を製造する方法が記載されているが、単相組織であるＮｉ－Ｔｉ金属間化合物とＴｉ$_{3}$Ａｌ とＴｉＡｌとの２相組織であるＴｉ－Ａｌ系金属間化合物とは組織が本質的に異なり、また前記公報には高融点のＴｉと低融点のＡｌとを素材としてＴｉ－Ａｌ系金属間化合物板を製造する点について記載、示唆されるところはない。

【０００７】本発明はかかる問題に鑑みなされたものであり、工業的生産方法として実用性に優れたＴｉ－Ａｌ系金属間化合物板の製造方法、および板面方向の機械的特性に優れたＴｉ－Ａｌ系金属間化合物板を提供することを目的とする。この目的は下記の発明によって達成される。

【０００８】
【課題を解決するための手段】本発明のＴｉ－Ａｌ系金属間化合物板の製造方法は、請求項１に記載したように、金属間化合物の主相がＴｉ$_{3}$Ａｌ とＴｉＡｌの２相組織となるようにＴｉからなるＴｉ層とＡｌからなるＡｌ層とが交互に積層された積層体を圧延接合により作製する圧延接合工程と、全圧下率を３％以上として連続的

あるいは間欠的に加圧しながらＡｌの融点未満の温度に加熱保持し前記Ｔｉ層のＴｉと前記Ａｌ層のＡｌとを反応させてAl_3Ti を生成させ、未反応のＴｉからなる残存Ｔｉ層と前記Al_3TiからなるAl_3Ti層とが積層された第１反応積層体を形成する第１固相拡散熱処理と、前記第１反応積層体を加熱保持してTi_3Al とＴｉＡｌとを主相とする２相組織を形成する第２固相拡散熱処理とを備える。

【０００９】この発明によると、圧延接合により積層体を作製するので、通常の圧延設備により表面積の大きい板状の積層材を容易に得ることができ、引いては第１固相拡散熱処理、第２固相拡散熱処理を施すことで大面積のＴｉ－Ａｌ系金属間化合物板を容易に製造することができる。ところで、Ａｌ層のＡｌとＴｉ層のＴｉとが反応してAl_3Ti を生成する際、カーケンドール効果による空隙（ボイド）のほか、Ｔｉ、Ａｌおよび Al_3Tiの各結晶構造の相違に基づき３％程度の体積減少が生じ、多量の空隙が発生し、著しい場合には剥離が生じる。このため、単にＡｌの融点未満の温度で固相拡散熱処理を行っても、空隙のために反応が抑制されて未反応のＡｌが残存するようになる。残存Ａｌがあると、第２固相拡散熱処理の際に、残存Ａｌが積層体から流出してＴｉ－Ａｌ系金属間化合物が得られない。本発明では、Al_3Tiを生成させる第１固相拡散熱処理の際に、積層体の体積率を３％以上縮小すべく全圧下率を３％以上、好ましくは５％以上として連続的あるいは間欠的に加圧しながら前記積層体をＡｌの融点未満の温度に加熱保持する。その結果、空隙の生成を抑制ないし防止しつつ、残存Ｔｉ層と Al_3Ti層とが積層された第１反応積層体を容易に形成することができる。この第１反応積層体にはＡｌが実質的に含まれないので、第１固相拡散熱処理後の第２固相拡散熱処理においてはＡｌの融点以上の高温に加熱保持することができ、 Ti_3Al相とＴｉＡｌ相とを主相とする２相組織からなるＴｉ－Ａｌ系金属間化合物板を容易かつ効率よく製造することができる。

【００１０】また、前記第２固相拡散熱処理は、請求項２に記載したように、前記第１反応積層体を８８２℃未満の温度に加熱保持して前記残存Ｔｉ層のＴｉと前記Al_3Ti層のAl_3Tiとを反応させて Ti_3AlとＴｉＡｌとを生成させ、このTi_3Alを主相とするTi_3Al層と前記ＴｉＡｌを主相とするＴｉＡｌ層とが積層された第２反応積層体を形成する熱処理とすることができる。この請求項２に記載した第２固相拡散熱処理によると、加熱保持温度が８８２℃未満であるため、残存Ｔｉ（αＴｉ）が結晶構造の全く異なるβＴｉに変態して成長することがない。したがって特定の結晶面の配向を板面にほぼ平行に保持したまま Ti_3Al層とＴｉＡｌ層とが層状に積層された構造の第２反応積層体を容易に得ることができる。この第２反応積層体は、層状構造を有し、各層の結晶配向が概ね板面に平行である故に良好な機械的性質を有するＴｉ－Ａｌ系金属間化合物板として用いることができる。

【００１１】また、請求項３に記載したように、前記第２反応積層体をαＴｉ単相温度域で加熱保持して前記第２反応積層体の各相をαＴｉ相に変態させた後冷却する第３固相拡散熱処理を行うことにより、組織中の空隙量を減少させることができる。また、板厚方向のほぼ全域にわたり板面にほぼ平行に配向した Ti_3Al／ＴｉＡｌラメラを主体とする Ti_3Al／ＴｉＡｌラメラ粒組織からなるＴｉ－Ａｌ系金属間化合物板を容易に得ることができる。この金属間化合物板は、その結晶構造の故に板面方向の機械的性質に優れる。

【００１２】また、請求項４に記載したように、前記第２反応積層体を Ti_3AlとＴｉＡｌとの共析変態点未満あるいはαＴｉとＴｉＡｌとの共存温度域の温度に加熱保持する第４固相拡散熱処理を施すことにより、結晶の配向や層状組織を保持しつつ、組織中の空隙を減少させた機械的性質の良好なＴｉ－Ａｌ系金属間化合物板を容易に得ることができる。

【００１３】また、前記第２固相拡散熱処理として、請求項５に記載したように、前記第１反応積層体をαＴｉ単相温度域で加熱保持して前記第１反応積層体の各相をαＴｉ相に変態させ、その後冷却することにより、残存Ｔｉがー 旦βＴｉに変態するため結晶の配向がランダム化し、従って共析変態により生成した Ti_3Al／ＴｉＡｌラメラの配向もランダム化するものの、 Ti_3Al／ＴｉＡｌラメラ結晶粒組織を有するＴｉ－Ａｌ系金属間化合物板を容易に製造することができる。

【００１４】また、請求項６に記載したように、前記積層体を中心層に対してＴｉ層あるいはＡｌ層が対称に配置された奇数層からなる構造とすることで、ＴｉとＡｌとの熱膨張率差に起因した熱変形を防止することができ、熱変形に起因した製造トラブルを防止して生産性を向上させることができる。この場合、最外層をＴｉ層とすることで、すべてのＡｌ層はＴｉ層によって挟持された状態となるので、第１固相拡散熱処理の際に全Ａｌ層のＡｌを無理なく反応させて Al_3Tiを生成させることができ、未反応Ａｌの残存を防止することができる。また、万一、未反応のＡｌが残存しても、高温熱処理の際にその流出を防止することができ、所期のＴｉ－Ａｌ系金属間化合物板の製造歩留まりを向上させることができる。

【００１５】本発明のＴｉ－Ａｌ系金属間化合物板は、請求項７に記載したように、板厚方向のほぼ全域が実質的に Ti_3Al／ＴｉＡｌラメラ粒組織からなり、前記ラメラ粒組織は板面にほぼ平行に配向した Ti_3Al／ＴｉＡｌラメラを主体とするものであるので、板面方向の機械的性質に優れる。

【００１６】また、本発明の他のＴｉ－Ａｌ系金属間化合物

合物板は、請求項８に記載したように、Ti_3Al相を主相とするTi_3Al層とＴｉＡｌ相を主相とするＴｉＡｌ層とが板厚方向にマクロ的に積層されたものであるので、板厚の内部が均質な従来の金属間化合物板に比して良好な機械的性質を備える。

【００１７】

【発明の実施の形態】以下、本発明のＴｉ−Ａｌ系金属間化合物板の製造方法およびこの製造方法によって得られた本発明にかかるＴｉ−Ａｌ系金属間化合物板について詳細に説明する。

【００１８】本発明の製造方法を実施するには、まず、圧延接合工程によりＴｉからなるＴｉ層とＡｌからなるＡｌ層とが交互に積層された積層体を作製する。前記圧延接合工程は、Ｔｉ薄板とＡｌ薄板とを適宜の枚数を重ねて圧延接合して複数層の積層素材を得て、さらにＴｉ層とＡｌ層とが交互に配置されるように前記積層素材を適宜数重ね合わせて圧延接合するものである。圧延接合は、冷間で行えばよく、１回の圧延当たりの圧下率は２０〜６０％、好ましくは２５〜５０％程度で行えばよい。圧延接合後は各層の接合強度を向上させるために、積層素材あるいはその接合材をアルゴンガス等の不活性ガス雰囲気中でＡｌの融点以下の温度、例えば６００℃程度で数分〜十数分程度の拡散焼鈍を行うようにするのがよい。

【００１９】前記積層体の各層の厚さは、層厚が薄すぎると圧延接合工程における後述の拡散焼鈍の際に燃焼反応が生じるようになるので、２μm 以上、好ましくは５μm以上にするのがよい。一方、厚すぎると後述する固相拡散熱処理に長時間を要し、工業的生産性を損なうようになるので、５０μm 以下、好ましくは３０μm 以下にするのがよい。なお、圧延接合工程の途中あるいは後に適宜冷間圧延を施すことによって、前記積層体の各層の厚さを調整することができる。

【００２０】前記積層体のＴｉ層、Ａｌ層の各層数は任意に設定することができるが、積層体の厚さ方向（積層方向）において Ti_3AlとＴｉＡｌとの２相組織が得られるようにＴｉ−Ａｌ組成を設定する必要がある。２相組織が得られる平均組成としては、at%でＴｉ：６５〜５２%、好ましくは６０〜５２%程度（残部Ａｌ）である。

【００２１】もっとも、前記積層体の層数は、好ましくは奇数とし、最外層にＴｉ層が来るようにするのがよい。このように、中心層を中心として厚さ方向に対称にＴｉ層、Ａｌ層を配置することで、後述の固相拡散熱処理の際に熱膨張率差に起因した反り等の熱変形を防止することができる。また、Ａｌ層は必ずＴｉ層に挟持されるので後述の第１固相拡散熱処理の際にＴｉと容易確実に反応するようになり、未反応のＡｌの残存を防止することができる。また、万一、未反応のＡｌが残存しても、高温熱処理の際にその流出を防止することができる。

【００２２】圧延接合工程により作製された積層体は、次に第１固相拡散熱処理が施される。この第１固相拡散熱処理は、Ａｌの融点未満の温度、好ましくは６３０〜５００℃、より好ましくは６２０〜５５０℃程度の温度で、４０min 〜１０ｈｒ程度保持して、前記積層体のＴｉ層のＴｉとＡｌ層のＡｌとを反応させて、基本的にＡｌが無くなるまで Al_3Tiを生成させる処理である。例えば、平均組成をＴｉ−４５at%Ａｌとする場合、下記式によって全ＡｌとＴｉとが反応して Al_3Tiが生成し、未反応のＴｉが残存する。結局、第１固相拡散熱処理によって、残存ＴｉからなるＴｉ層とAl_3Ti相からなるAl_3Ti層とが積層した第１反応積層体が得られる。

$$55Ti+45Al \rightarrow 40Ti+15Al_3Ti$$

【００２３】ところで、Ｔｉ、Ａｌ、Al_3Ti の結晶構造から格子定数を考慮して体積変化を計算すると、Al_3Ti が生成する際の反応では体積減少が起こり、前記例示組成の場合では、その減少率は３．２%になる。カーケンドール効果のほか前記体積変化により残存Ｔｉ層に沿って多量の空隙（ボイド）が形成され、前記拡散反応が阻止されるようになる。また、著しい場合には層の剥離をも招来する。前記体積減少率３．２%はＴｉ−４５at%Ａｌの場合であるが、Ti_3Al とＴｉＡｌとの２相組織が得られる成分範囲については概ね３〜４％程度の空隙が形成されると考えられる。なお、前記体積率の減少量は、Ａｌ層、Ｔｉ層は厚さ方向に積層されているので、板厚方向の減少率として考えることができる。

【００２４】このため、本発明では、第１固相拡散熱処理の際に、Al_3Ti が生成する際に形成される空隙を排除すべく、全圧下率を３%以上、好ましくは５%以上として積層体を連続的あるいは間欠的に圧下する。これによって空隙が排除され、Al_3Ti の生成反応が促進される。前記第１固相拡散熱処理の際の圧下方法としては、熱処理の途中に積層体に数%ないし十数%程度の軽圧下を数回以上、好ましくは数分ないし数十分間隔で付与してもよく、あるいは積層体に１０〜７０ＭＰａ程度の圧縮荷重を常時付加するようにしてもよい。なお、圧下率（%）は（板厚減少量）／（初期板厚）×１００を意味する。

【００２５】次に、前記第１固相拡散熱処理によって得られた第１反応積層体に対してTi_3Al とＴｉＡｌとを主相とする２相組織を形成する第２固相拡散熱処理を施す。先の組成例で説明すると、この第２固相拡散熱処理における反応は下記式にて表すことができる。なお、この反応では相全体の体積は２%程度の増加になる。

$$40Ti+15Al_3Ti \rightarrow 5Ti_3Al+40TiAl$$

【００２６】この第２固相拡散熱処理として、前記第１

反応積層体を８８２℃未満の温度に加熱保持して前記残存Ｔｉ層のＴｉと前記Al_3Ti層のAl_3Tiとを反応させてTi_3AlとＴｉＡｌとを生成させ、このTi_3Alを主相とするTi_3Al層と前記ＴｉＡｌを主相とするＴｉＡｌ層とが積層された第２反応積層体を形成する熱処理を採ることができる。

【００２７】この熱処理の特徴は、図１のＴｉ－Ａｌ二元系状態図中に記したＴ１線から理解されるように、残存Ｔｉ（αＴｉ）をβＴｉに変態させることなく、前記２相組織を得ることができる点にある。αＴｉから結晶構造が著しく異なるβＴｉに変態すると、結晶の配向が乱れ、また粒成長を起こして粗大化するが、この熱処理によれば結晶粒を粗大化させることなく、αＴｉの結晶の配向（多くは板面にほぼ平行になっている。）を維持したまま２相組織を得ることができる。しかも、主に残存Ｔｉのある部分からTi_3Al相が生成しAl_3Tiの部分からＡｌＴｉ相を生成するため、マクロ的にTi_3Al層とＴｉＡｌ層とが積層した層状構造の第２反応積層体を得ることができる。すなわち、この積層体は、その層状構造と結晶面の配向のために、板面方向の機械的性質が良好となる。なお、この第２反応積層体は本発明のＴｉ－Ａｌ系金属間化合物の一態様をなすものである。

【００２８】前記第２固相拡散熱処理の加熱保持は、酸化防止のために真空中で行うことが好ましい。また、加熱保持温度は、αＴｉがβＴｉに変態しないようにするためには８８２℃未満に設定すればよいが、処理の安定性を考慮すると好ましくは８７０℃以下、より好ましくは８６０℃以下にするのがよい。また、固相拡散の効率を考慮すると、８００℃以上に設定することが好ましい。加熱保持時間は、８００℃以上の温度で加熱保持する場合、１５～２５ｈｒ程度とすればよい。

【００２９】本発明においては、前記第２固相拡散熱処理によって得られた第２反応積層体をさらにαＴｉ単相温度域で加熱保持して前記第２反応積層体の各相を一旦αＴｉ相に変態させ、その後冷却する第３固相拡散熱処理を施すことができる。加熱温度は要はαＴｉ単相組織が得られる温度であればよく、その上限は液相が生じることなく、またβＴｉ相が生じない温度であればよい。加熱保持は酸化防止のために真空中で行うことが好ましく、また保持時間は、全組織をαＴｉ単相組織に変態させるとともに結晶粒の粗大化が生じないように、５～１５ｈｒ程度が好ましい。

【００３０】この第３固相拡散熱処理を施すことで、図１のＴ２線で示すように、一旦αＴｉに変態した後、共析変態点（１１１８℃）を通過する際に共析変態を起こして、Ti_3Al とＴｉＡｌとがミクロ的に層状のラメラ粒組織となり、しかも多くのラメラの配向が板面にほぼ平行になるため、板面方向に優れた機械的性質を有するＴｉ－Ａｌ系金属間化合物板を得ることができる。また、第２反応積層体に中間反応生成物（例えば、Al_2Ti ）や未反応Ｔｉが一部残存する場合においても、これらを完全にTi_3Al やＴｉＡｌに反応させることができ、また残存した空隙をより減少ないし消失させることができ、金属間化合物の品質を向上させることができる。なお、過共析組成の場合、αＴｉから温度の低下に従って初析ＴｉＡｌ（γ）が生成するが、このγ相はラメラ状に析出してくることが本発明者によって確かめられている。

【００３１】前記第２反応積層体に対する熱処理としては、上記の第３固相拡散熱処理に限らず、図１のＴ３線で示すように第２反応積層体をTi_3Al とＴｉＡｌとの共析変態点（１１１８℃）未満あるいはαＴｉとＴｉＡｌ（γ）との共存温度域の温度で加熱保持する第４固相拡散熱処理を施すこともできる。加熱保持温度は、反応の促進からは高い方がよいため、下限は９６０℃、より望ましくは１０００℃程度とすることが好ましい。もっとも、αＴｉとγとの共存温度域は高温領域まで及ぶので、結晶粒の粗大化防止、マクロ的積層構造の維持の観点からは共析変態点未満の温度で処理することが好ましい。この第４固相拡散熱処理における加熱保持も真空中で行うことが好ましく、また保持時間は結晶粒の粗大化が生じないように１０～２５ｈｒ程度が好ましい。この第４固相拡散熱処理によって、第２反応積層体の層状構造や結晶の板面方向の配向をある程度維持したまま、中間反応生成物等が生じることが無く、空隙も減少ないし消失した高品質の金属間化合物を得ることができる。

【００３２】上記第３、第４固相拡散熱処理は、第２固相拡散熱処理を施して得られたTi_3Al相 とＴｉＡｌ相とを主相とする第２反応積層体に対する熱処理を示すものである。これに対し、第２固相拡散熱処理としては、第１反応積層体を８８２℃未満の温度で加熱保持することなく、Ti_3Al とＴｉＡｌとの共析変態点超のαＴｉ単相温度域の温度で加熱保持後冷却する熱処理（α熱処理と呼ぶ。）としてもよく、あるいは第１反応積層体を共析変態点未満あるいはαＴｉとγとの共存温度域の温度で加熱保持後冷却する熱処理（β熱処理と呼ぶ。）としてもよい。対象とする反応積層体は異なるが、熱処理方法の考え方としては、α熱処理は前記第３固相拡散熱処理に対応し、β熱処理は前記第４固相拡散熱処理に対応した熱処理であり、その加熱温度、時間については前記第３、第４固相拡散熱処理と同様に設定することができる。もっとも、α熱処理の場合、Al_3Ti が液相にならないように１３８７℃未満の温度で加熱保持する必要がある。

【００３３】前記α熱処理、β熱処理の場合、結晶粒が粗大化し、残存Ｔｉ（αＴｉ相）がβＴｉ相に一旦変態するため板面に平行な結晶の配向も損なわれることになるが、Ti_3Al とＴｉＡｌとを主相とする２相組織を有するＴｉ－Ａｌ系金属間化合物板を容易に得ることが

できる。

【0034】以下、実施例によって本発明をさらに説明するが、本発明はかかる実施例によって限定的に解釈されるものではない。

【0035】

【実施例】実施例1

(1) 圧延接合工程

純Ｔｉ板と純Ａｌ板の表面を金属ブラシを用いて粗く削った後、削った面どうしを重ね合わせて圧下率３０％にて冷間圧延を行い、その後、Ｔｉ層とＡｌ層とが圧接された複合材をアルゴン雰囲気中で６００℃、１０分間の拡散焼鈍を行い、両層が密着した２層素材を得た。この２層素材をＴｉ層とＡｌ層とが交互に重なるように３枚重ね合わせて圧延接合し、拡散焼鈍することで６層素材を得た。最終的にＴｉ層とＡｌ層とが交互に重ね合わされ、両最外層がＴｉ層となるように５５層からなる積層体を作製した。その際、積層体の平均組成がＴｉ−４５．５at％Ａｌとなるように板厚をＴｉ板：０．２５mm、Ａｌ板：０．２０mmとした。また、最終的に得られた積層体のＴｉ層の平均厚さは１７μm 、Ａｌ層の平均厚さは１３μm であった。

【0036】(2) 第１固相拡散熱処理

前記５５層の積層体に対して、６００℃で合計１ｈｒ加熱保持した。この際、図2に示すように、圧下率１０〜３％の熱間圧延を１０分間隔で６回行った。この熱処理によって得られた第１反応積層体のマクロ的断面組織をＳＥＭにより観察した。その結果を図3に示す。ＥＰＭＡによる分析からＡｌは完全に反応していることが確かめられたので、観察される層はAl_3TiからなるAl_3Ti層と未反応のＴｉからなる残存Ｔｉ層であることがわかった。また、板面方向に伸びる線状の空隙が認められた。

【0037】(3) 第２固相拡散熱処理

前記第１反応積層体を真空中で８５０℃にて２０ｈｒ保持した。この際、積層体に熱変形（反り）が生じるのを防止するため、ステンレス片の重りをセラミック板を介して第１反応積層体に載置した。前記積層体にかかる圧縮応力は３ｋＰａ程度であった。前記セラミック板は、重りと第１反応積層体との反応を防ぐためのものである。この熱処理によって得られた第２反応積層体の断面組織をＳＥＭにより観察した。その結果を図4に示す。第２反応積層体は、主にＴｉＡｌ相と Ti_3Al相とが厚さ方向に積層された構造を示しているが、微量のＴｌ、Al_2Tiが残存しており、また微細な空隙が観察された。

【0038】(4) 第３固相拡散熱処理、第４固相拡散熱処理

前記第２反応積層体を真空中で１３００℃にて１０ｈｒ保持し、徐冷した（第3固相拡散熱処理）。この熱処理によって得られた金属間化合物板のミクロ的断面組織をＳＥＭにより観察した。その結果を図5に示す。図5に示されるように、ラメラが板面にほぼ平行に配向したＴｉＡｌ(γ)／Ti_3Al(α_2)ラメラ組織粒が観察された。また、空隙については、第２反応積層体に比して線状の空隙は無くなり、空隙量も少なかった。一方、前記第２反応積層体を真空中で１１００℃にて１０ｈｒ保持後、徐冷した（第4固相拡散熱処理）。この熱処理によって得られた金属間化合物板のマクロ的断面組織をＳＥＭにより観察した。この観察結果を図6に示す。図6より、板厚方向の積層構造は第２反応積層体よりも崩れたものの、マクロ的には全体として積層構造が維持されていた。また、空隙については、表面近傍には比較的多く観察されるものの、内部には微細な空隙しか観察されなかった。

【0039】実施例2

(1) 圧延接合工程

前記実施例１と同様にして、５５層からなる積層体を作製した。

【0040】(2) 第１固相拡散熱処理

前記５５層の積層体に対して、真空中で５０ＭＰａの圧縮応力を付加して６００℃にて５ｈｒ加熱保持した。

【0041】(3) 第２固相拡散熱処理

引き続いて、第１反応積層体を８５０℃にて２０ｈｒ保持した。この際、圧縮応力を５０ＭＰａから１０ＭＰａに減少させ、その後１０ＭＰａで保持した。平均圧縮応力は２２ＭＰａであった。この結果得られた第２反応積層体は、マクロ的には層状構造をしており、空隙はほとんど観察されず、局所的にわずかな線状の空隙が見られる程度であった。また、ＥＰＭＡ分析により、観察される相は、主相がTi_3AlとＴｉＡｌとであったが、一部Ｔｉ、Al_3Ti、Al_2Ti も認められ、第１実施例の第２反応積層体に比して反応が遅かった。これは、ＴｉとTi_3Al の２相が生成する際に生じる体積増加を圧縮荷重によって抑制したためと推測される。従って、圧縮荷重の付加は、第１固相拡散熱処理の段階で十分であり、第２固相拡散熱処理においては積層体の反り発生を防止する程度の軽荷重の付加に止めることが有効である。

【0042】(4) 第３固相拡散熱処理

前記第２反応積層体を真空中で１３００℃にて１０ｈｒ保持後、徐冷した。得られた積層体の断面をＳＥＭにて組織観察した結果、フルラメラ組織が観察され、空隙量も非常に微量であり、健全性に優れた金属間化合物板が得られらた。

【0043】

【発明の効果】本発明の製造方法によれば、通常の圧延設備を用いて、Ｔｉ−Ａｌ系金属間化合物板を容易かつ効率的に製造することができ、工業的製造方法として優れる。また、本発明のＴｉ−Ａｌ系金属間化合物板は、板面方向の機械的性質が良好であり、実用性に優れる。

【図面の簡単な説明】

【図1】Ｔｉ−Ａｌ二元系状態図である。

【図2】実施例１の第１固相拡散熱処理における積層体に対する圧下状態を示す温度−時間図である。

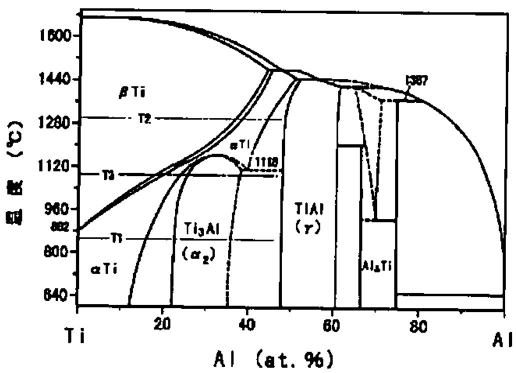
【図3】実施例１の第１固相拡散熱処理後の反応積層体のマクロ的断面組織を示す図面代用ＳＥＭ組織写真である。

【図4】実施例１の第２固相拡散熱処理後の反応積層体のマクロ的断面組織を示す図面代用ＳＥＭ組織写真である。

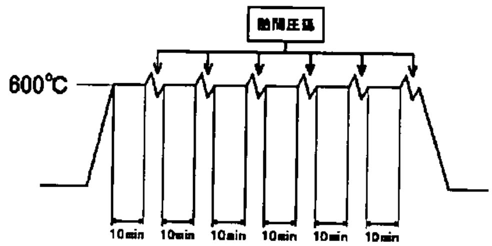
【図5】実施例１の第３固相拡散熱処理後の反応積層体のミクロ的断面組織を示す図面代用ＳＥＭ組織写真である。

【図6】実施例１の第４固相拡散熱処理後の反応積層体のマクロ的断面組織を示す図面代用ＳＥＭ組織写真である。

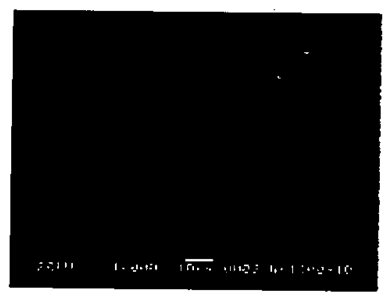
【図1】



【図2】

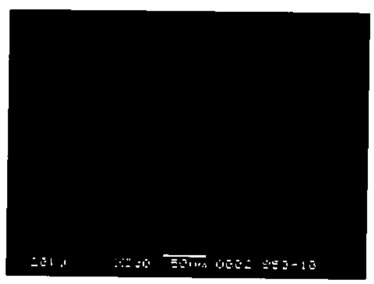


【図5】



【図3】

【図4】



【図6】

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		686			686B
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